

Reductive Amination of 5-Methoxytryptamine (Mexamine) to 5-MeO-DMT with NaBH₄ and Paraformaldehyde: An Eyewitness Testimonial Of A Partially Successful 50g Run.

Preface

In this synthesis the starting material is 5-Methoxytryptamine, which reacts with formaldehyde in methanol solution to form an imine intermediate which is then reduced with NaBH₄ (adds hydrogen). After two "cycles" 5-MeO-NMT (methyltryptamine) should form, after another two "cycles", you should have 5-MeO-DMT, you could do another cycle, but it would be a wise choice to invest into a TLC system so you could make a more informed decision instead of rolling dice during the procedure. This method allows one to use fairly accessible chems, no longer do you need to use sodium cyanoborohydride. Low temperature is key, once you start to understand every individual part of the synthesis, the yield should improve and the procedure moves along faster.

In no way is this a final guide, it is more of a rough draft, a report on a semi-successful run, but it should help the reader to get an understanding of the overall process. From this particular report a beautiful crystalline product was obtained albeit in a poor yield, however there were not one or two mishaps in my bid.

Hope to follow up with another report but not of 5-MeO-DMT. Now let's get cookin', and let's save some toads while we're at it! (or trees in the case of DMT)

Chem info:

5-Methoxytryptamine (5-MeO-T, O-Methylserotonin, mexamine) C₁₁H₁₄N₂O, Mol. weight: **190.246 g/mol**; Melting Point: **119 – 123 °C**; Slightly soluble in methanol, chloroform
Appearance: white to light yellow to light orange powder.

5-MeO-DMT (5-methoxy-N,N-dimethyltryptamine) C₁₃H₁₈N₂O, Mol. weight: **218.3 g/mol**, Melting Point (freebase): **66-67 °C**, 67-68 °C, 69-70 °C (various sources). pKa: 9.3, Soluble in: Chloroform, ether, DCM, acetone, methanol, ethanol, insoluble in water. Hexane/ naphtha (60 – 80 °C, heavy?) /Pet ether are commonly used to crystallize it and also heptane.

Off-white crystals, could be white initially then yellows quickly in presence of air.

Formaldehyde (methanal) CH₂O, Mol. weight: **30.026 g/mol**, colorless gas

paraformaldehyde (CH₂O)_n is formaldehyde in a polymer, in a chain of 8 -100 units long, one must depolymerize to formaldehyde before using it, calculate molar mass like formaldehyde.

White powder, or granules, mild odor of formaldehyde

formalin is an aqueous solution of formaldehyde and could be stabilized by methanol. Sold in various concentrations, 10, 20, 30%, etc. or a max of 36 - 38 % (by weight), calculate for the molar mass of formaldehyde in solution. Density: 1.08 – 1.09 g/cm³

Sodium Borohydride (NaBH₄), Mol. weight: **37.83 g/mol**, white powder/ crystals, hygroscopic, forms a cake exposed to air

Acetic acid (glacial acetic acid, GAA) Mol. weight: **60.052 g/mol**, density of liquid form: 1.049 g/ cm³, colorless liquid

Sodium (Na): **22.99 g/mol**, 0.9688 g/cm³, silvery white metallic solid

Methanol (CH₃OH, abbreviated as MeOH): boiling point: 64.7 °C, density: 0.792 g/ cm³, colorless liquid

Dichloromethane (DCM): boiling point: 39.6 °C, density: 1.3266 g/ cm³, colorless liquid
Hexane (n-hexane): boiling point: 68.5 – 69.1 °C, density: 0.6606 g/ cm³, colorless liquid

Part 1: Purification of discoloured, and impure tryptamines

Preface

This is a procedure that a vendor suggested, and it could be key to get a better yield out of your starting tryptamine or 5-MeO-T. If you are planning a synthesis I would suggest to not skip this part if the material has a darker colour. And it could also be utilized to purify other tryptamines, finished actives.

As an example starting from **31.235 g** of a darker 5-MeO-T, stored in a freezer, but colour is far from white. Calculate as **0.16418 mol**.

1) Use a 1.05 mol. equivalent of acetic acid to 5-MeO-T or 0.1724 mol. or 10.352 g or **9.87 mL**. Suck it up with a 10 mL pipette and mix with 250 mL dH₂O to make a dilute solution. Soak the mexamine with a few hundred mLs of water, mix with a spoon or with magnetic stirring. Add the dilute acid to the mexamine, and keep adding more water until almost everything is dissolved, in the procedure decide to add a few more drops of acetic acid to lower the pH to 5.5 (check with a pH meter). Use about **600 mL water to 31 g 5-MeO-T**. (You could use more water to fully dissolve everything, but just).

Remember that if you store glacial acetic acid (pure acetic acid) in a cold room, there might be solids floating, it could be frozen over, then lightly warm the acetic acid to fully dissolve it.

2) Transfer to a 2 L separatory funnel and pour in about 250 mL DCM, some black mass appears on the walls of the sep. funnel, and the DCM takes on a darker ruby color. There are black floaters on the bottom of the aqueous layer.

3) Discard both the bottom DCM layer, and try to get rid of as much sludge, and floaters as possible without pouring out too much of the aq. layer. You will not keep the DCM, store in your waste bottle to discard later.

4) Drain the top layer with the 5-MeO-T directly into a pyrex dish on a hotplate, heat at 60 - 80 °C to gently bubble off any remaining DCM, now the water layer has a darker colour but as I remember the DCM is darker.

5) Check that no DCM remains by looking for small bubbles evolving as the DCM boils, or not recommended – by checking for a DCM odor. Set in a ring stand a wide glass funnel and fold a large medium speed filter into a flute, pour from the pyrex dish through the filter to gravity filter into a beaker.

6) Prepare a sol. of maybe 7 g sodium hydroxide (NaOH) in 250 mL water. Wait for both the dark orange aq. layer and the NaOH sol. to cool a bit.

7) Under stirring, start pouring in the lye solution. At around 9.9 pH most of the product seems to have precipitated out, I keep adding lye water until a pH of 11.9, although maybe there is no need to add above 10.5. Should have used most of the lye water, if not whip up a bit more. Remember, pour some water into the beaker, then drop in the NaOH under stirring, then add water to the desired volume when preparing a solution of NaOH.

8) Vacuum filter the aq. layer making sure before pouring onto the Büchner funnel that the filter paper has a good seal. Transfer to a crystallizing dish, fan dry for a few hours. The material dries fairly quickly. Move it around with a big ass spoon, sift into a fine powder.

Now we should have something that looks much nicer. Might be almost white, maybe slightly tan with no smell. That dark slime won't harm the reaction anymore.

From two runs have these yields:

Starting material: **25 g**, end up with **21.745 g**, starting material **31.235 g**, end up with **29.589 g**. So you do lose a few grams purifying, but it is written in a few places, that it is very important to have a clean starting material, especially in the case of 5-MeO-T.

Before we get cookin' for real, before we start flippin' them pancakes, I would like to note, that if you can't get a hold of 5-MeO-T, it is possible to start with **melatonin** and synthesize 5-MeO-T from that. There are a few simple methods of hydrolysis, with n-butanol and NaOH, or tosylic acid and NaOH. It adds a few more steps, maybe an extra day to the synthesis, but the procedures do not seem difficult at all. Then the synthesis is even more OTC. Melatonin is not the cheapest material, but sometimes it is possible to find it sold as a bulk powder.

And **tryptamine** can be prepared by the decarboxylation of **L-tryptophan**. It is a very cheap material, and can be bought in bulk as a supplement. The decarb proceeds in turpentine with a ketone catalyst such as **spearmint oil** (mostly carvone). There is also this method that is microwave assisted:

<https://www.dmt-nexus.me/forum/default.aspx?g=posts&t=96211>

Part 2: Preparation of methanolic formaldehyde solution and the reaction in a dry ice/ acetone bath.

Preface

As one can apply the process of purification in Part 1 to a wide variety of tryptamines, so one can run this exact procedure with tryptamine, only accounting for small differences in stoichiometry to yield a product of DMT. In fact it is possible to substitute formaldehyde for acetaldehyde in the synthesis of DMT to end up with DET. Start from 5-MeO-Tryptamine, run two cycles first with acetone, then two cycles with formaldehyde, you should have the wonderful 5-MeO-MiPT, with tryptamine, MiPT. It also seems like a pathway to DPT, start with tryptamine, mix with propionaldehyde.

Again, little difference in procedure whether starting from tryptamine or 5-MeO-Tryptamine. And I did do a smaller run with tryptamine, did make DMT, however I did not know how to extract it properly in the work-up, and as well did not end up with crystal DMT but with this sticky DMT honey. So it could be that 5-MeO-DMT is easier to make if you are after crystals, and not a melted and sticky product.

Next time I will try to make DMT with modifications and see if I get a good yield with a crystalline product. But stock up on both T (seems to be much cheaper), and 5-MeO-T, and make both products. Mix up the spice!

1) First we need to prepare a solution of formaldehyde in methanol. This avoids the use of any water in the reaction, which should help it along, as well as prevent side-reactions. The equilibrium of the reaction should proceed forward more when water is not introduced. We will make a solution of formaldehyde in methanol that is 30%. It might be possible to heat paraformaldehyde in methanol until it depolymerizes and dissolves, but it is also possible to use sodium or lithium metal to dissolve it.

During the reaction we will need 5x the molar amount of formaldehyde as 5-MeO-T. So if we start from **51.334 g 5-MeO-Tryptamine (0.27 mol)** will need to use **1.35 mol** or **40.535 g formaldehyde** during the reaction. Let's use a bit more and take **42 g of paraformaldehyde, mix with 140 mL of dry methanol** on it, this should make a 30% solution. Note: it would be wise to don a mask when handling paraformaldehyde, but it seemed mostly benign when I was around it, it certainly did not have a smell that formaldehyde is reputed to have. Don't sniff the MeOH too much, it's not meow.

Now you might have received sodium metal vacuum wrapped in plastic or maybe dipped in paraffin.

Sodium metal is very reactive towards water, and quite a bit towards methanol, but less so. As well it might be better to limit the exposure time to air. With a scalpel I cut off a piece of sodium metal in the paraffin, wipe it with a paper towel, weigh and then quickly transfer to a petri dish with a layer of toluene. Drop it in there so it doesn't react.

To dissolve this much paraformaldehyde you need about **190 - 200 mg**. That is what should work. I used 180 mg for 40.535 g paraformaldehyde which was slightly short. The sodium helps to depolymerize the PF, it also should form some sodium methoxide, I could be wrong on this. A regular procedure to make formaldehyde stock solution utilizes NaOH, but we are working without water so use elemental sodium metal.

Place the paraformaldehyde + MeOH mixture under magnetic spinning, and drop in the sodium, maybe 1/3 at a time. There is an exotherm and the metal dances and fizzes on the surface of the methanol, but slowly the paraformaldehyde starts to dissolve, as it is converted to formaldehyde in methanol, keep on adding that shiny metallic armor until all that PF goes bye-bye. We want a beautiful clear solution of MeOH.

Side-quest: Dry MeOH? Yes, it might be best to dry your MeOH before the reaction day. You can do this by dropping in a 20% w/v amount of molecular sieves 3Å into your MeOH container, maybe a day or two before crunch time. Drying molecular sieves? Well that is a long story for another bedtime. It could be done in a microwave, but you also need a vacuum to store the sieves quickly once the cool. Honestly, I do not think the sieves I used to dry the MeOH were fully activated. I would explore more traditional methods of drying MeOH next time.

After all the Na has been added, it might take up to some 30 minutes for the solution to go clear, but at these amounts the PF should dissolve sooner or later.

2) This reaction needs to run cool. How will we do that? Well for smaller amounts it could be enough to make an ice-water-salt bath, but I think the larger additions of NaBH_4 and the larger exotherms at this 50g amount necessitate the use of dry ice. So we will make an acetone/ dry ice bath.

This necessitates a trip to your friendly local dry ice stockist. Just don't forget to grab that beer cooler before heading out. And make sure you get them nightcrawlers still squirming about on the bottom out of the way. Lyophilized creepy crawlers haven't made it yet to Parisian brasseries. After your purchase of -78°C CO_2 you have to be committed to the procedure. Yes sir I am! The dry ice sublimates away, if left overnight even in the beer cooler in winter a portion will be gone. So plan out the reaction for the same day, or the next. 3 – 5 kg will be enough of this super ice, the rest you can put into pumpkins for a spooky effect.

So, into a large glass dish, maybe 250 mm wide, 100+ mm tall, add about 1 kg of dry ice, and carefully spray, wet, the dry ice with acetone first. Use cheap hardware store acetone. Go slowly, there will be fizzing and hissing, when the dry ice is partially submerged, maybe you can pour in the acetone but do it slow. The last time during the procedure I am writing about, I was not careful at all with the dry ice and poured a bunch of acetone on top. The dry ice went ballistic and rattled so much I thought it was dear old grandma shaking in her nightgown like a skeleton. The dry ice cracked and ruined the glass dish, had to switch to one that was too large, in consequence, could not cool sufficiently, and I believe yields suffered immensely. So even handling the dry ice in the incorrect way can have an outcome on your results. Also, I had no experience and no clue what to expect during addition. Here is what smarter people than me have to say on the issue:

"Slowly adding acetone to dry ice will minimize the amount of dry ice you need to handle. Alternatively, you must slowly add dry ice to the acetone or the large volumes of carbon dioxide produced will cause rapid bubbling." Now I know.

The temperature in the bath should drop very low, well below -30°C , beyond the range of any human thermometer.

The glass bath will radiate heat or rather heat transfer will occur rapidly. So insulation is needed. A dewar flask is used in these sorts of situations or some glass dish in a glass dish with perlite filling contraption, but there is another "elegant" solution: a 1 meter x 1 meter Styrofoam insulation block. So get one of those, maybe one that is 50 mm thick, cut it in half and cut out a circle the size of your wish with a hacksaw. But watch out, soon your apartment will be like a scene from that awful period in history, The Great Styrofoambowl. Okies with hungry children traversing a bleak plain with Styrofoam tumbleweed rolling by in the stiff carbon dioxide wind...

3) Now we can mix up the 5-MeO-T with a 17x amount in mL of MeOH or 870 mL for the 51.334 g (0.27 mol.) 5-MeO-T we have on hand. The substrate should dissolve easily with such a proportion. It should be possible to lower the proportion to 15x, maybe one could do 200 g in 3 L MeOH...

As well it might be a smart move to prepare your NaBH_4 maybe even your formaldehyde solution at this point.

If the reader recalls, mention was made of "cycles" in the beginning, the plan for this synthesis are 4 rounds of formaldehyde addition and sodium borohydride additions, with warming to destroy the NaBH₄ then cooling again before beginning another round.

To be precise, before the 1st round, cool to however low it takes so that the NaBH₄ addition does not heat up so much as to allow a Pictet–Spengler reaction to occur. Various temps are reported, but try to stay below -5 °C when product is reacting. -25 °C as the starting point could be a good idea.

The plan once sufficiently cooled is to add in a 2 mol. eq. of formaldehyde from the 5-MeO-T amount: 0.54 mol or 16.214 g or 54 mL of the solution. Then let it react 15 minutes at the cold temperature, but really best below -20 °C at least, and then add a 0.6 mol. amount of NaBH₄ to the amount of formaldehyde.

Calculate the amount of formaldehyde in relation to the 5-MeO-T, and the NaBH₄ to the amount of formaldehyde.

So add in 0.324 mol of NaBH₄ but don't do it in a single pour, pour in bit by bit watching the temperature closely so it does not exceed -5 °C. Add dry ice as needed. Once all of the NaBH₄ is poured in, react for 1 hour in the cold (I would say at least -20°C, or freezer temp, -25).

4) After an hour place the 2 L three-neck round bottom flask into a heating mantle and heat gently to 30 °C, the methanol should begin to bubble, as hydrogen is released and the NaBH₄ disintegrates into borates. Cool to -20 °C in preparation for another addition of formaldehyde and then NaBH₄. That is the general pattern.

So,

1st addition: 2 mol formaldehyde to 5-MeO-T, **0.6 mol NaBH₄** to *formaldehyde* amount

2nd 3rd, and 4th additions: 1 mol formaldehyde to the starting 5-MeO-T amount, and **0.6 mol NaBH₄** to the *formaldehyde* amount.

This is not the only way of running the reaction, and it could be better to run the reaction with no heating between additions, would be easier as well. Just make sure it is always cold. Could utilize two addition funnels with your three neck flask, like Hamilton Morris did, but then you would need to dissolve the NaBH₄ in water. Or you could do small additions of NaBH₄, but more of them, and heat only at the end. Need to do more testing to figure out just which method works best. As well, you do not need to add so much dry ice, it does not need to be extremely cold, the reaction slows and sodium borohydride is not as soluble in MeOH in extreme arctic conditions. A mild Canadian winter is what we want.

In fact when I ran it with tryptamine, notwithstanding an extraction failure, I think the reaction went well without heating in between additions. Of course the reaction was over quicker. Also, if you heat up and there is still unreacted amine, there is a greater chance for the Pictet–Spengler reaction to take place. Beta-carbolines might be formed. So I will try to run it once again but all at -20 (except right after NaBH₄ addition when temps temporarily rise).

What would help immensely in running this particular reaction is a TLC system (thin layer chromatography). And I will certainly invest in one the next time around. Look into a portable UV Lamp 365 nm/254 nm, and some GF254 Silica Gel plates 25 x 75 mm.

This is a good guide on TLC, and a good guide for other lab techniques:

[https://chem.libretexts.org/Courses/SUNY_Oneonta/Chem_221%3A_Organic_Chemistry_I_\(Bennett\)/2%3ALab_Textbook_\(Nichols\)/02%3A_Chromatography/2.03%3A_Thin_Layer_Chromatography_\(TLC\)](https://chem.libretexts.org/Courses/SUNY_Oneonta/Chem_221%3A_Organic_Chemistry_I_(Bennett)/2%3ALab_Textbook_(Nichols)/02%3A_Chromatography/2.03%3A_Thin_Layer_Chromatography_(TLC))

For tryptamines specifically here is a system that works well:

from Sawdust and Honey, The Vespiary:

I used the same 1mm/80mm capillary tubes I use for melting points. With the compound concentration I had (50mmol in 250ml methanol), the mixture itself was a bit too concentrated. So what I did was take up 2-3 drops of the reaction mixture and dissolve it in 3-5ml methanol. A TLC starting line was drawn with a pencil (be careful not to scrape down the silica) and a very small, 1 (only one!) spot was done of each reaction mixture and solution of tryptamine in methanol (conc. very small too, I can't lie to you, I just eyeballed a couple tens of mg of tryptamine and dissolved it in 10ml of methanol and used it as a standard in all my TLCs). You should use a 254nm UV lamp, don't bother with KMnO₄ or iodine. UV is MUCH, MUCH cleaner and non-intrusive. Just make sure that your plates are covered with F254 or something similar. They should be greenish, while the surroundings in UV are violet/blue. You'll see whether the spot is concentrated enough if the spot is well-visible and small in diameter. You will also need to adjust your concentrations a bit with experiments, if you're getting smeared results then the concentration is too small.

The eluent used was 10:0.5:0.5:0.25 volumetrically ethyl acetate:methanol (ethanol works too):n-hexane:25% ammonia.

Realistically I recommend doing 10 drops of ethyl acetate to 1 drop of everything else. Or you can just make up some of the eluent, it's generally useful for tryptamines. N-alkylated tryptamines tend to be less polar and therefore climb higher on the plates.

<https://www.thevespiary.org/talk/index.php?topic=16470.msg54205305#msg54205305>

With TLC it would be possible then to determine if there is unreacted tryptamine left, and whether to proceed with a 5th cycle.

5) To recap calculating by weight and volume: for the **1st cycle** add **54 mL 30% formaldehyde in MeOH**, and **12.257 g NaBH₄**, for the **2nd – 4th cycles** add **27 mL 30% formaldehyde in MeOH**, and **6.129 g NaBH₄** each cycle.

During the 3rd and 4th cycles had to switch to an ice-water-salt bath. And after NaBH₄ additions it was impossible to keep temperatures low enough. Temps spike to 12 °C. As well after the 3rd and 4th additions, once it reacts at the colder temp for 1 hour, during warming of the reaction mixture not much bubbling occurred like it did after the 1st and 2nd cycles. Could the NaBH₄ been destroyed earlier?

I heat at 35 °C for 20 minutes, but other procedures state to heat at **50 °C for 30 minutes**.

Wiki states: "Complete decomposition of a methanol solution requires nearly 90 min at 20 °C."

6) Gravity filter through medium filter paper into a 2 L round bottom flask and go to sleep.

Also, I ran the reaction in a 2 L three-neck round bottom flask but used magnetic stirring, for larger amounts it could be useful to utilize overhead stirring. Did not run this in an inert atmosphere, but you could easily fill a balloon on a syringe with argon gas, and fit on a rubber septum. However the additions would require removal of the septum, unless some sort of a solid addition funnel is used.

Part 3: Work-up of the reaction and extraction of 5-MeO-DMT freebase

1) Now we shall see what we can do. The plan is to vacuum filter off the methanol until only a thick dark sticky mixture of potentially unreacted products, maybe tar, possibly beta-carbolines, and our product is left. To protect the product from degrading or further, a vacuum is used to lower the distillation temp. And you need a good vacuum, it would be good to distill the methanol at 25 °C, however I had too many catastrophes at this stage too count, had violent boiling, even switched between two pumps, the temperature definitely ran too high, and after distilling off 1/3 of the methanol I gave up. Don't be me, get a pump that is cable of a deep vacuum, cool the condenser with cold water, and place the receiving flask in cold water to further aid condensation, it could be wise to use short-path distillation or shorter condenser. But above all a good vacuum source is crucial.

2) Prepare a 400 mL 5% potassium carbonate (K_2CO_3) solution. It could be possible to skip this step, and once the methanol is distilled off, just to pour in water. This is to raise the pH to above 12. Starting pH already basic at above 10.6, so I am not sure about this step.

Into the thicker reddish brown methanol, pour K_2CO_3 solution until pH rises above 12, not all 400 mL is used. White particulae fall out, mixture becomes milky red-brown. I could have used a 10% solution. There is a total of 625 mL.

Now the plan is to extract this layer with DCM, but the remaining methanol could have really screwed things up.

3) Pour the reaction mixture into a 2 L sep. funnel, taking care to fish out the magnetic stirrer. Will extract 5 times with 70 mL DCM each time. After thorough mixing of the DCM with the aq. layer, the DCM forms an emulsion and forms trouble. Small drops of DCM are trapped in the upper layer, everything is too thick, and the DCM is too slow to sink to the bottom. Worry not, salt is here. If faced with an emulsion, return everything to sep. funnel and prepare a brine solution. Maybe 100 mL. A concentrated solution of NaCl in water, about 35g/ 100 mL. Just make sure your salt is reagent grade or kosher. Now mix in the brine and gently mix the layers together. The salt will aid in separation, it will still be slow, but it should separate now. The brine reduces surface tension or something, attracts water.

It is reputed that chloroform will not form emulsions in this case, and it could be a better choice when making these compounds. Also DMT in particular reacts with DCM forming potentially quaternary ammonium salt byproducts, albeit slowly. see:

Code Select

<https://pubs.acs.org/doi/10.1021/acsomega.8b00507>

When working with chloroform there is the draw-back that it is toxic to the human chemist not just fish.

The first extract was very thick, the DCM pulled out a bunch of goodies. Follow up with a total of 6 more times, and the DCM still had a yellow tint during the 7th extraction. Each extract was about 75 mL each, so I used too much DCM, something was wrong here.

Now the DCM will be very dark, but it will also have lots of water, so we use magnesium sulfate to clear it up.

4) In a 2 L beaker under magnetic stirring dump in about 10 g anhydrous magnesium sulfate

(MgSO₄), watch as the solution majestically clears up, keep adding and mixing in smaller portions watching so that the MgSO₄ becomes free flowing and stops clumping, this means that all of the water has been absorbed. Continue mixing for maybe 10 minutes more. Again, the DCM layer will still have a reddish tint, but it should be clear now. Filter the DCM into a 1 L round bottom flask to vacuum distill off.

DCM boils easily but is difficult to condense, so again a good vacuum goes a long way, but I also found that running ice cold water through the condenser and setting the receiving flask in a dish with ice water also helps.

The other option is to pour into a hotplate and evaporate off the DCM with gentle heating and a fan blowing. But this is rather a bit of DCM to evaporate off, so do this only in a fume hood. A wasteful method, and as well the product is very sticky, it will be a pain to scrape it all off from a plate.

5) Scrape the sticky stuff into a 1 L beaker, or 500 mL, if it is difficult to work with, one can heat it up and it will turn more viscous. If one has vacuum distilled off the DCM, it should be in a round bottom flask, just waiting for your next move.

6) Weigh the goop taring from the known weight of a 500 mL beaker and it is about 59.68 g. Now how much is pure 5-MeO-DMT? We need to get it out either with hexane or heptane. Chose hexane if heptane is too expensive, but go with heptane as it is much less toxic. You can re-use it for many extractions. So it might be good to invest in a 5 L bottle of the highest quality heptane for your chemistry endeavors. But who knows, maybe you have access to cheap heptane, lucky you then.

Now the procedure becomes in a way similar to DMT extractions from mimosa root bark. And it is a way to remind you of your humble beginnings. A reminder of how far you've gone. Or a too gentle prod to remind you that in fact you've gone way too far son. But mom, just let me cook this DMT one last time!

Ok, how much hexane or heptane to use? I did not get this part correct last time so I used some info from DMT-Nexus as a guide:

solubility in boiling n-Hexane (69 °C)

1.9 g in 100 ml

19.0 g in 1 l

Let's assume a yield for the reaction of 70% (things could have gone better) this works out to 0.189 mol 5-MeO-DMT freebase or 41.26 g, so this amount should dissolve in 2170 mL boiling n-hexane (or roughly the same for heptane) in boiling naptha the solubility is much greater.

<https://www.dmt-nexus.me/forum/default.aspx?g=posts&m=1090269#post1090269>

7) Try and extract with all of the hexane I have first. Will do 5 extractions of 310 mL each as I have a bit over 1.5 L. To carry out the extraction you need to set your sludge in the 500 mL beaker on a hotplate/ magnetic stirrer and heat to 80 °C or so, separately in a 2 L beaker set your hexane to boil.

Pour the boiling hexane into the beaker with your hot sludge, let them mix for a bit with magnetic stirring, simultaneously you can thrash the sludge with a spoon to pick it up from the

bottom more. After some mixing I rapidly decant the hexane while it is boiling into a pyrex dish, repeat the procedure 5 or so times making sure not to get any sludge from the bottom when decanting.

During the extractions the hexane did not seem to take on any colour, but there was product there.

After some cooling the hexane becomes cloudy, milky white, and after maybe 30 minutes the first crystals should start forming. It is nice to observe your labour coming to fruition this way. Just wear a mask when your face is 5 cm from the hexane for prolonged periods.

As well the sludge layer in the beaker should start shrinking, I suppose if the synthesis went well, the sludge layer should mostly disappear, should be left with a very small amount that will be junk to discard.

As for the hexane/ heptane, save that. If you did not have enough for the extraction, you can re-use it after you decant from the plate.

The hexane cools, beautiful crystal grow, I cover the plate, and eventually stick it in a freezer.

8) Continue the extraction with naptha, but use the wrong kind. One with a high boiling point that I believe has quite a bit of toluene (or other aromatics), it seems to extract too much, and has a horrific odor. Do only one extraction, as it cools can see that the naptha layer is discoloured. Place this naptha plate in the freezer as well.

The hexane plate is full of white crystals, snowflakes, longer spider veins, while the naptha plate is a sludge on the bottom, I decant the naptha in disgust to a waste container. Scrape the sludge and add it to the beaker with the material I was extracting from.

And do another run with boiling hexane, place into a second plate.

9) After some time in the freezer, decant the hexane through filter paper, fan dry some, and then scrape the plate with a razor blade. In total from plate 1 collect 12.96 g and from plate 2, somewhere around 2.5 g. If using the amount of hexane that I used for plate 1, I should have been able to capture 28.5 g, so plate 2 should have none, but there was some product, maybe I still don't know how to extract. Continue saving the hexane as it looks clean enough.

10) Next will use a plastic crystallizing dish for DMT, maybe product doesn't stick so much to it, as it is a chore to scrape these large glass dishes. Remember it might look cool on TV, but Video Killed The Radio Star. Also, seems that the longer it spends in the freezer the more that the crystals spread on a hard layer on the dish. Or maybe small amounts of product on a large surface are no good.

With DMT the plastic crystallizing dish (lab grade PP plastic) method might very well help to get any potential honey product off easier. Then scrape with a plastic card, or pop off by flexing the plastic.

11) With the product dry, I do a **melting point** test using a thiele tube, fill it with paraffin and heat the loop with a primus stove on low heat, watch very closely, and heat very slowly once past 50 °C, something happens at **62 °C** it could be that the sample sinters, then it melts maybe at 64

°C and is a puddle at **68 °C**. So I would say the melting point test is passed, the sample matches the melting point for 5-MeO-DMT freebase.

12) Well that is that, a **yield** somewhere over 15.18 g from 51.33g. A 100% yield would have been 58.9 g, so I have a paltry **30%**. So this run was rather wasteful of the 5-MeO-T. But this is enough 5-MeO-DMT freebase for all of the neo-shamans in my village to last a lifetime. Still that methanol screwed me over, and the naptha, and the dry ice bath, arghhh!!!!

In memoriam

This is just one of many ways to go about the work-up. It is possible to use ethyl acetate to extract, and then distill concentrate and then the 5-MeO-DMT should solidify. That is how Hamilton Morris went about it on Vice. My intuition tells me something would be messy about that with my crappy gear. I would try a chloroform + heptane combo, or continue using DCM with heptane. Hexane and heptane are very selective, they should not pick up anything besides the DMT or 5-MeO-DMT, but I am not certain, will need to read up more and really run the reaction with TLC.

Regarding DMT , there is this quote from Nick Sand:

The DMT required distillation and recrystallization to obtain pure product. Distillation would be set up on the laboratory lattice (Figure 4.6) and 1 or 2 kg would be processed at a time. A kilogram of DMT dissolved in 2L ether would recrystallize when added to 8L hexane. Rhomboid crystals would spontaneously form in the mother liquor.

(<https://www.erowid.org/archive/rhodium/chemistry/tryptamine2dmf.html>)

Fin

Some pics

Album URL:

<http://dumpliwoard5qsrssroni7bdiishealhky4snigbzfmzcquwo3kml4id.onion/album/c66c8f920128f868>

Sodium in toluene in a petri dish, and in the beaker undissolved paraformaldehyde in methanol
9c3c001439b11383.png

2 L three neck flask with the reaction mixture after the reaction I believe
b58075ef5c708f48.png

DCM causes an emulsion
45a2fd804b81f65e.png

2nd extraction, clearer
e0b86b10677f1fc6.png

Big ass shards, still with hexane, xtals stuck firm to bottom
f7dd5f19070d0a85.png

Crystal macro
25cbb0e51d2f81bf.png

Thiele tube
e667882cce26fdcd.png

Wanted to add: substance was bioassayed and it is very strong, even at 2mg the effects are strong. Not very pleasant at low doses. Made a vape 50 mg/ 0.5 mL, still powerful. Think I prefer plain DMT.

Post-script: The heating is not needed between cycles afterall, use a stoichiometric amount of NaBH_4 , everything happens after the last cycle. This makes the reaction quicker and easier.